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THE VAPOR PRESSURE OF LEAD CHLORIDE

BY

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THE VAPOR PRESSURE OF LEAD CHLORIDE.

By E. D. EASTMAN and L. H. DUSCHAK.

INTRODUCTION.

During the past few years interest has been revived in the possibility of treating certain complex ores, especially those of lead, zinc, silver, and copper, by a process involving a chloridizing roast, and subsequent volatilization of part or all of the valuable metallic constituents. In order to make a technical study of this process, a knowledge of certain fundamental data, such as the vapor pressures of the chlorides of the metals involved is essential. There is a decided lack of such information in the literature. The Bureau of Mines has been making an experimental study of the volatilization process in order to determine its possibilities and limitations and has also undertaken to determine the vapor pressures of the important metallic compounds concerned in the process. The vapor pressure of lead chloride was the first to be investigated.

GENERAL EXPERIMENTAL METHOD.

In principle the method used by the bureau is the familiar one of determining the weight of the substance under investigation that is required to saturate a measured amount of inert gas at known and constant temperatures and pressures.

DESCRIPTION OF APPARATUS.

In practical detail, the apparatus is similar in some respects to that of Welch and Duschak[a] and that of von Wartenberg.[b]

As shown in figure 1 (see also Pls. I, *A* and *B*, and II, *A*) it consists of a tube *a*, closed at one end, and containing a boat, *b*, of the material to be studied. The part containing the boat is separated from the rest of the tube to form a vaporizing chamber by means of a second closely fitting tube *c*. Through holes in the end of the latter tube there project side by side over the boat a tube *d*, in which the

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[a] Welch, H. V., and Duschak, L. H., Vapor pressure of arsenic trioxide: Tech. Paper 81, Bureau of Mines, 1915, p. 6.

[b] Von Wartenberg, H., Über Metalldampfdrucke: Ztschr. Elektrochem, Bd. 19, 1913, p. 482.

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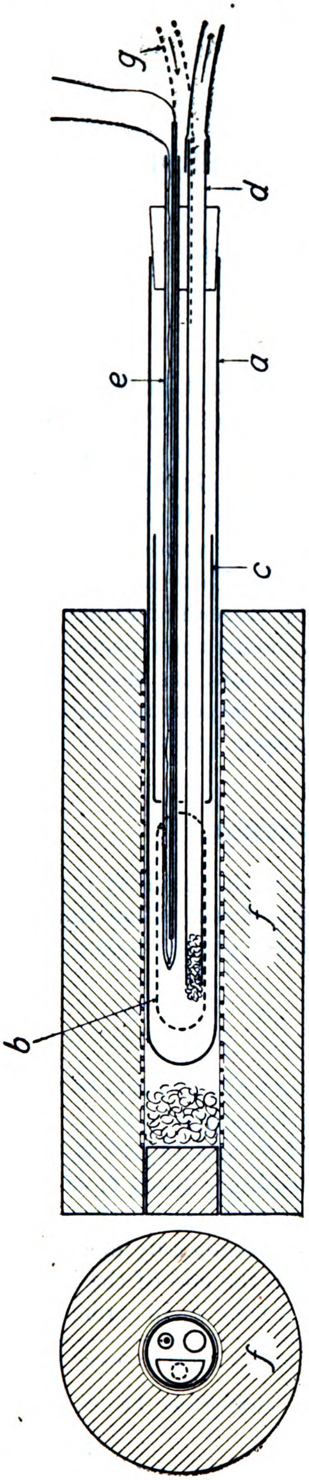


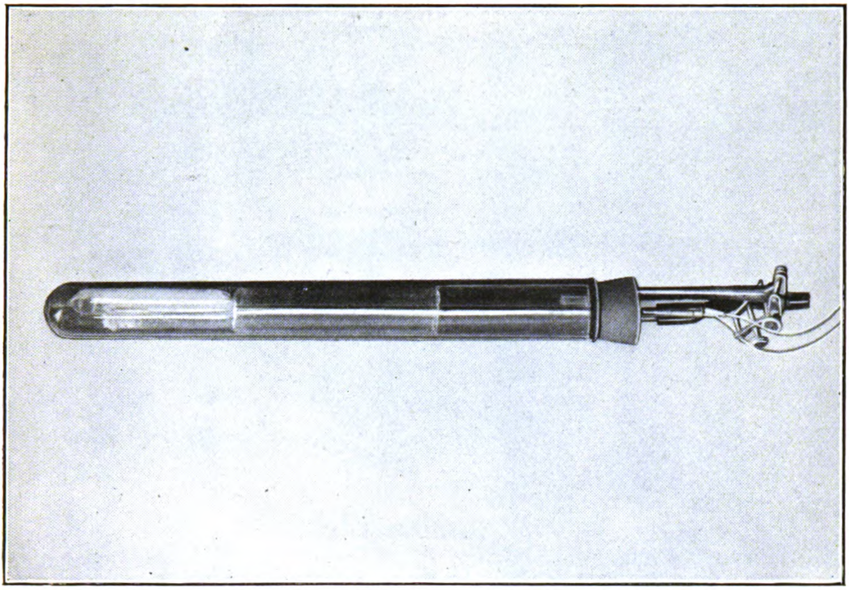
FIGURE 1.—Sections of electric furnace and apparatus for saturating gas stream with lead chloride, vapor.

vapor is condensed, and a tube, *e*, inclosing a thermocouple. Another tube, *g*, extends just through the rubber stopper which closes tube *a*. The apparatus is heated in an electrical tube-furnace *f*, regulated by hand.

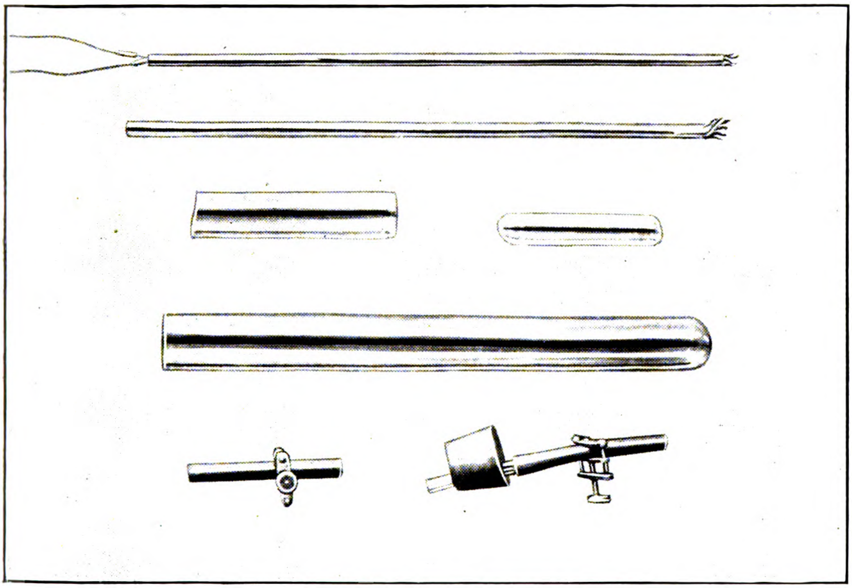
PROCEDURE.

The method of manipulation is as follows: During the heating period, the gas to be used (maintained by a mercury escape valve at constant pressure slightly greater than atmospheric) is passed into the apparatus through the tube *d*, and allowed to waste through the tube *g*, until the air and moisture originally present are swept out. When this has been accomplished, and approximate regulation of the furnace at the desired temperature is attained, the direction of flow through the apparatus is reversed, the gas entering at *g*. Convection within the tube *a*, with the attendant effect upon the degree of saturation and constancy of temperature, is reduced by the restricted area of passage between tubes *a* and *c*. The gas enters the vaporizing chamber through this annular space and passes over the material in the boat before reaching the opening of the condensing tube. A porous plug at this point serves as a filter to remove droplets of suspended liquid. As the mixture of gases, after passing the plug, approaches the colder end of the tube the vapor condenses. The permanent gas goes on and is collected in an aspirator bottle, the water displaced being measured in graduated flasks.

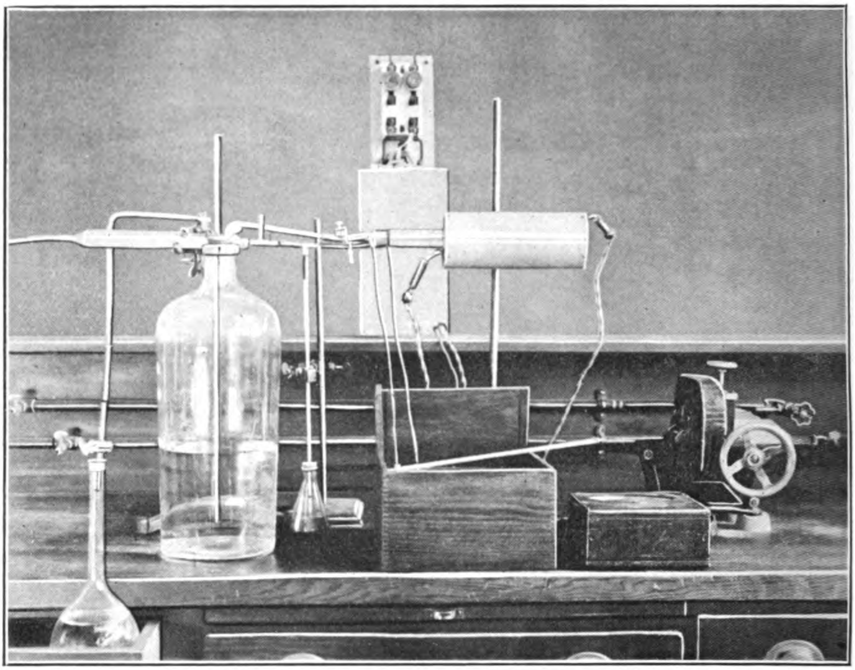
The gas in the aspirator is brought to the pressure at which the run is to be made by connecting the aspirator to the tube *g* for a short time at the beginning of the experiment. When



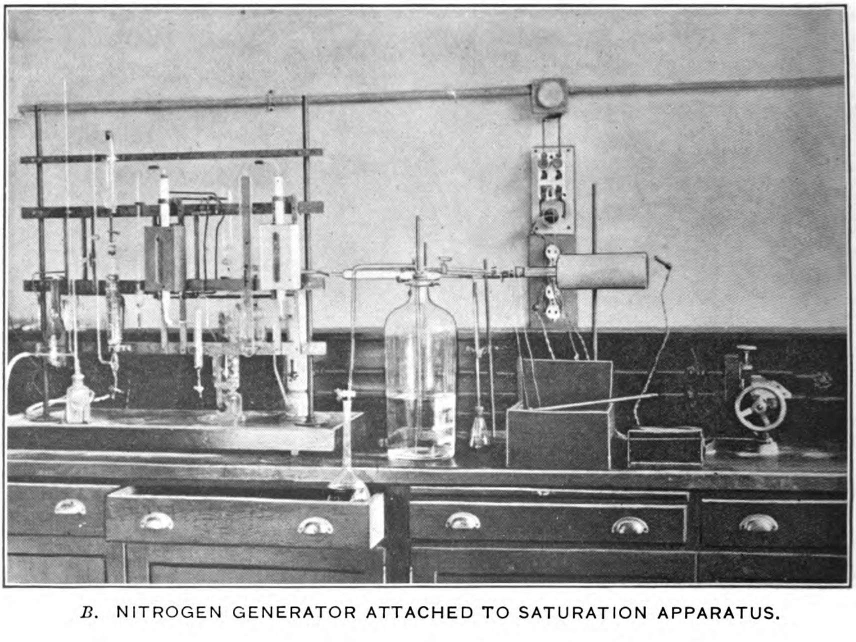
A. SATURATION TUBE ASSEMBLED.



B. PARTS OF SATURATION TUBE.



A. SATURATION APPARATUS COMPLETELY ASSEMBLED.



B. NITROGEN GENERATOR ATTACHED TO SATURATION APPARATUS.

small volumes are to be measured, the gas may be advantageously collected directly in inverted graduated flasks by displacement of water. The weight of the substance carried as vapor by the gas and deposited in the condenser is determined after the experiment by an appropriate volumetric or gravimetric method.

The principal sources of experimental error inherent in the method, aside from those in the temperature and volume measurements and in the analysis, are the lack of constancy and uniformity of the temperature throughout the vaporizer and the possible failure to obtain saturation of the gas with vapor. The latter error may be considered to have been eliminated if successive runs at the same temperature with considerably varying rates of flow of gas do not show systematic variations in the results. If for any reason the rates can not be made slow enough to accomplish this, extrapolation to zero rate may be used. Steps that may be taken to control other errors of general and special character will be mentioned later.

DETAILS OF THE EXPERIMENTS.

The lead chloride used was prepared by recrystallization from solution in normal hydrochloric acid. Two lots of the commercial "C. P." salt from different manufacturers were found to be interchangeable with the recrystallized material so far as the vapor pressures were concerned.

Preliminary experiments showed that air containing water vapor reacted readily with lead chloride, hydrochloric acid being obtained even at temperatures below the melting point (500° C.), and chlorine at higher temperatures. When dry air was passed over the chloride at temperatures as low as 550° C., appreciable amounts of chlorine were detected in the issuing gas. In the vapor-pressure measurements, therefore, nitrogen was always used.

The nitrogen was obtained by the method of Lupton.[a] The generator, which was designed and built in the bureau laboratory, is shown at the left in Plate II, *B*. Air is bubbled through aqueous ammonia, and then passed over electrically heated copper gauze in a clear silica tube. The operation is so regulated that all but the first portion of the copper gauze remains bright, thereby indicating the presence of a slight excess of ammonia. Careful tests of the apparatus have shown that under these conditions no oxides of nitrogen are formed, the issuing gas carrying only ammonia and a small trace of hydrogen. The ammonia is removed by passing the gas through dilute sulphuric acid and the hydrogen by passage through a second electrically heated silica tube containing copper oxide. The gas is then passed successively through concentrated sulphuric acid and over soda lime and phosphorus pentoxide.

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[a] Lupton, Sidney, Laboratory notes; preparation of nitrogen: Chem. News, vol. 33, 1876, p. 90.

For temperatures up to 650° C. the vaporizing and condensing tubes were of pyrex glass; above this temperature silica was used. The boat containing the lead chloride was of silica in all the experiments, and the plug (see fig. 1, p. 6) was of fluffy asbestos. Some other material, such as quartz wool, would have been more suitable, as the asbestos may have given off some water at the higher temperatures, although it had been previously ignited.

The outer tube *a* of the apparatus, which was about 25 centimeters long and 2.5 centimeters in internal diameter, fitted closely into an electrical tube furnace, the heating coils of which extended several centimeters beyond the ends of the vaporizing chamber. The furnace was closed with a cement plug, the space between this plug and the end of the tube *a* being loosely packed with asbestos. The temperature of the furnace was regulated and held constant by adjustment of an impedance coil in series. The temperature was read at uniform short intervals, the greatest deviation from the average usually being 3° C., though in some cases momentary deviations of 5° C. occurred. There was usually a small temperature gradient in the vaporizing chamber. The variation, however, was never found to exceed 1° per centimeter for a distance of 5 centimeters over the boat. There was evidence of distillation of the lead chloride toward the ends of the tube, droplets of the liquid being found there and on the end of the partition tube after runs. Opportunity for diffusion of vapor into the condenser after the flow of gas was stopped was avoided by immediate removal, either of the condensing tube alone or of the entire apparatus from the furnace.

Temperatures were measured with a platinum and platinum-rhodium thermocouple made by Heraeus. Small Marquardt tubes were used for insulation of the wires in the silica-containing tube. The voltage was read with a "Northrup millivolter," a null method instrument, and also with a Leeds and Northrup thermocouple potentiometer, readings with the two instruments agreeing throughout the range used to within 0.03 millivolt, an amount not greatly in excess of the error of setting and reading. The standard curve of Adams[a] for the Le Chatelier couple was made use of in converting voltages to temperatures, the cold-end correction being made from the table in Burgess' and Le Chatelier's[b] book on "The Measurement of High Temperatures."

The boiling point for sulphur and melting point for sodium chloride as found with the thermocouple were in exact agreement with this scale.

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[a] Adams, L. H., Calibration curves for copper-constantin and platinum-platinrhodium thermocouples: Jour. Am. Chem. Soc., vol. 36, 1914, p. 71.

[b] Burgess, G. K., and Le Chatelier, H., The measurement of high temperatures, 3d ed., 1912, p. 156.

The weight of lead chloride recovered was determined by steaming out the condenser tube and titrating for chloride by the Volhard method. Tenth and hundredth normal solutions of $AgNO_3$ and KSCN were used, the following procedure being uniformly adhered to. A small excess of $AgNO_3$ solution was added to the $PbCl_2$ solution, and the AgCl formed was coagulated by boiling. The solution was then cooled to the temperature of the room, 1 c.c. of 10 per cent $Fe_2(SO_4)_3$ and 2.5 c.c. of concentrated HNO_3 were added for each 25 c.c. of solution, and the solution titrated with KSCN. The $AgNO_3$ solutions were standardized with recrystallized $PbCl_2$ prepared for the vapor pressure measurements. On account of the fact that AgSCN is less soluble than AgCl, the end point, when a small amount of $PbCl_2$ was determined with the 0.01 normal solution, was not permanent. In standardization, however, the deviations of individual results from the average was not greater than 1 per cent for the 0.01 normal solution and 0.3 per cent for the 0.1 normal solution.

A fresh charge of lead chloride was used for each experiment. At the higher temperatures the material in the boat, but not that in the condenser, was often found to be colored gray at the end of the run. Analyses of the residues showed 99 per cent $PbCl_2$ for runs at 850° C.; 99.5 per cent for runs at 800°; and 99.8 per cent at 700°. In these runs varying amounts of HCl were found on titrating the water over which the nitrogen was collected (by displacement from inverted flasks). The largest amount found was equivalent to 2 per cent of the total volume of gas passed. The formation of HCl may have been due to water being liberated from the asbestos plug, as previously suggested, or to the presence of a small amount of hydrogen in the nitrogen. The gray color of the residues might indicate the latter, as the correct explanation. The errors from this cause are partly compensating, and in any event are believed not to equal those from other sources.

That there was no loss of lead chloride as fume in the gas coming from the condensing tube would seem to be established by the fact that lead could never be detected in the glass wool plugs which were frequently used in the outlet of the condenser. These plugs absorbed some HCl from the gas and consequently gave tests for chloride, but never for lead. Their use was finally discontinued.

RESULTS AND CALCULATIONS.

The results of all experiments which were completed, and the magnitudes of the quantities directly measured are shown in Table 1.

TABLE 1.—*Results of experiments.*

Serial number of experiment.	Temperature.	Weight of $PbCl_2$ recovered.	Volume of N_2 passed (room condition).	Rate of flow of N_2.	Vapor pressure
	°C.	*mg.*	*c.c.*	*c.c. per minute.*	*mm. of Hg. at 0° C.*
40	502	15.1	5,530	18	0.186
36	507	12.8	4,000	21	.218
34	530	18.6	3,500	23	.357
29	549	8.6	1,000	13	.58
30	550	10.1	1,000	6	.69
25	551	16.5	1,520	20	.74
26	551	19.1	2,005	22	.66
27	552	18.2	2,000	10	.63
28	552	19.6	2,000	12	.67
37	567	31.2	2,000	22	1.07
31	577	35.4	1,500	15	1.58
32	590	31.0	1,000	11	2.14
22	600	40.9	1,000	10	2.84
21	601	53.7	1,000	12	2.92
33	625	39.5	502	9	5.3
18	649	62.4	500	7	8.6
19	649	59.8	500	8	8.1
35	678	46.0	203	8	15.2
15	707	184	500	8	21.7
16	707	195	500	7	26.0
8	752	417	500	6	52.7
13	752	251	302	6	53.4
14	752	170	200	4.2	53.6
12	798	408	200	12	117
10	799	420	200	7	121
11	799	388	200	5	112
17	846	447	100	2.3	217
20	847	481	100	4.2	228
BP	945	…………	…………	…………	754
39	445	2.7	22,000	55	0.0033

As an illustration of the way in which the vapor pressure values given in Table 1 were obtained, the calculations for one of the runs at 752° C. will be indicated. In run 13, 0.2510 gram of 0.000904 mol. of lead chloride were recovered. The volume of nitrogen passed was 0.3015 liter, measured over water at 21.0° C. and 754 mm. Calculating the volume of dry gas under standard conditions and dividing by 22.4 gives the number of mol. of nitrogen as 0.01209. The mol. fraction of lead chloride vapor in the gases withdrawn from the vaporizing tube was, therefore,

$$\frac{0.000904}{0.000904+0.01209}=0.0696.$$

The mol. fraction times the total pressure gives, of course, the corresponding partial pressure, which in this instance is also the vapor pressure. The total pressure[a] in the vaporizer in this instance was 768 mm., giving as the vapor pressure $0.0696\times768=53.4$ mm. This calculation is based on the assumption

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[a] The total pressure is the atmospheric pressure plus the slight positive pressure within the apparatus. (See p. 6.)

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that the gases involved are perfect and that the molecular weight of lead chloride vapor corresponds to the simple formula. The latter assumption is supported by the vapor density measurements of Scott.[a]

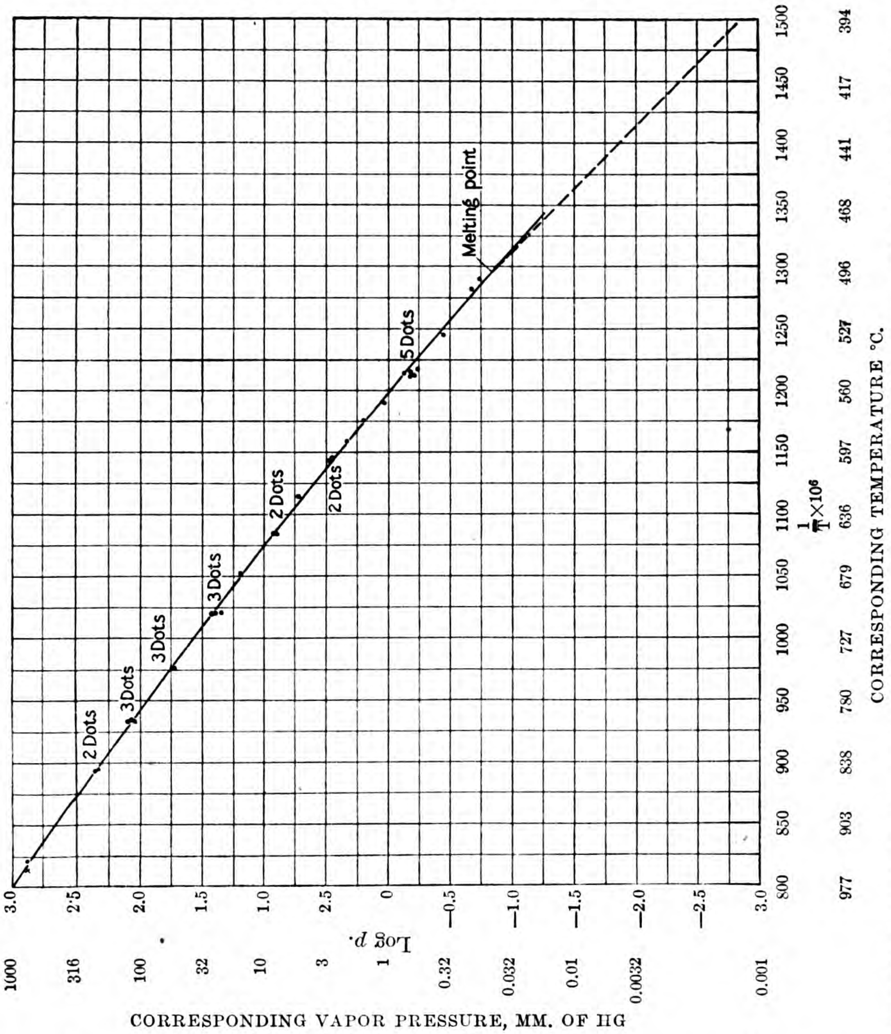

FIGURE 2.—Curve showing common logarithm of vapor pressure plotted as function of the reciprocal of the absolute temperature.

The vapor pressure data of Table 1 are shown graphically in figure 2, the familiar method being adopted of plotting the common logarithm of the pressure in millimeters of mercury, against the reciprocal of the absolute temperature.

In addition, the boiling point 956° C., reported by Weber[b] and that determined in his laboratory, are represented by the cross and the near-by dot, respectively. The boiling-point determination was made in an atmosphere of N_2 in a silica test tube 2.5 cm. in diameter. The tube extended into the heating coil of the furnace a distance of 2.5 cm., and was insulated with asbestos above this point. The $PbCl_2$ filled the bottom of the tube to a depth of 4.0 cm. Boiling was very smooth and temperatures constant. However, the temperature varied with varing positions of the thermocouple, as indicated in the following: With the junction of the couple at the bottom of the liquid the temperature recorded was 954° C.; junction just below the surface of liquid, 948°, 8 mm. above the surface, 945°; 22 mm. above the surface, 942°. There was no discoloration of the $PbCl_2$ after the run. The temperature represented on the diagram is 945°, and the pressure 754 mm.

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[a] Scott, Alexander, On some vapor densities at high temperatures: Proc. Roy. Soc. Edinburgh, vol. 14, 1887, p. 410.

[b] Weber, O. H., Uber die Anderungen der freien Energie bei geschmolzenen Halogenverbindungen einiger Metalle: Ztschr. anorg. Chem., Bd. 21, 1899, p. 305.

The continuous curve of the figure has been drawn as best representing the experimental points for the liquid lead chloride, and the dotted line shows values calculated for the solid lead chloride as explained below. From the smooth curves the vapor pressures have been taken at 25° intervals between 400° and 950° C., and appear in the second column of Table 2, calculated values being understood. In the third column of the table are shown the weights, calculated from the vapor-pressure data, of lead chloride required to saturate one cubic meter of gas at the various temperatures. The weights given in the fourth column are those necessary to saturate one cubic meter of gas, measured at 0° C., and heated at constant pressure to the temperatures indicated. These values are convenient for calculating the weight of $PbCl_2$ required to saturate any given volume of air or furnace gases.

The vapor pressures of the solid salt were evaluated with the aid of the Clausius equation. If any two vapor pressures of the liquid are represented by p_1 and p_2, T_1 and T_2 being the corresponding absolute temperatures, Q the molal heat of vaporization, and R (1.98 calories) the gas constant, the equation is

$$\ln \frac{p_2}{p_1} = \frac{Q}{R}\left(\frac{1}{T_1} - \frac{1}{T_2}\right) \qquad (1)$$

The formula is exact if the vapor is a perfect gas, and if Q and the total pressure on the liquid are constant between the temperatures involved. When these conditions are fulfilled, the graph of $\ln p$ or $\log p$ against $\frac{1}{T}$ is a straight line. The curvature in the lead-chloride curve is, no doubt, to be attributed chiefly to rather rapidly changing values of Q, readily accounted for on the basis of changing ionization or polymerization, or both, in the liquid lead chloride. The curve as drawn is, in this instance, a straight line in the interval just above the melting point. Hence, the calculation of Q by substituting in equation 1 vapor pressures from the curve gives the value of Q at that temperature; then, from the known value of the heat of fusion, the heat of sublimation at the melting point may be calculated. Equation 1 applies to the solid, if the heat of vaporization is replaced by the heat of sublimation. The latter quantity may be assumed to be constant, thus permitting the calculation of vapor pressures below the melting point. The single value for the solid, which is necessary for this calculation, was obtained from the temperature of the liquid at the melting point, where the two curves must intersect.

Table 2.—*Vapor pressures of lead chloride and weights of unit volumes of saturated vapor.*

[Values in italic are calculated and relate to the solid salt.]

Temperature.	Vapor pressure.	Weight of 1 cubic meter of saturated vapor.	Weight of $PbCl_2$ required to saturate 1 cubic meter of gas measured at 0° C. and heated to indicated temperatures, pressure remaining constant.
° C.	*mm. of Hg. at ° C.*	*grams.*	*grams.*
400	*0.00174*	*0.0116*	*0.0284*
425	*.0058*	*.0371*	*.095*
450	*.0178*	*.110*	*.291*
475	*.051*	*.304*	*.83*
500	.141	.81	2.30
525	.326	1.82	5.3
550	.71	3.84	11.6
575	1.45	7.6	23.7
600	2.82	14.4	46.2
625	5.0	24.9	82.2
650	8.7	41.9	144
675	14.1	66	235
700	22.4	103	377
725	35.5	158	608
750	53	232	930
775	80	338	1,460
800	116	481	2,235
825	166	670	3,470
850	231	920	5,420
875	320	1,240	9,030
900	433	1,650	16,430
925	600	2,240	....
950	770	2,820	....

Two determinations of the melting point (in pyrex tubes in atmospheres of CO_2 and N_2, respectively) gave 493° C. and 497° C. The mean, 495° C., average with the better determination in the literature, gives 498° C. The vapor pressure was taken, from the curve at this point, to be 0.137 mm. and the heat of vaporization calculated as 40,600 calories. Goodwin and Kalmus[a] report 5,150 calories as the molal heat of fusion of lead chloride, their value being chosen for calculation rather than the older and apparently less accurate one of 5,800 calories obtained by Ehrhardt.[a] The heat of sublimation then is 45,750 calories.

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[a] Goodwin, H. M., and Kalmus, H. T., On the latent heat of fusion and the specific heat of salts in the solid and liquid state: Phys. Review, vol. 28, January, 1909, p. 1.

The "smoothed" results of Table 2 are believed to be correct within 5 per cent above 550° C. For definition of the curve with accuracy in the lower range, more numerous data would be desirable, but were deemed unnecessary for the purposes in view. While the error in the values for the "solid" curve as a consequence of the uncertainty in the "liquid" curve, can not be estimated, these figures are probably to be regarded as maximal, with considerable deviations possible. They are, however, considered to be more reliable than the single experimental point, obtained by the method of Welch and Duschak[b] at the boiling point of sulphur. This point is given in the last line of Table 1, and, as may be seen from fig. 2, falls much below the point on the calculated curve at this temperature, the values being 0.0033 mm. and 0.0145 mm., respectively.

SUMMARY.

1. A method for the measurement of vapor pressures at high temperatures is described.
2. The details of its application and the experimental results for lead chloride at temperatures between 500° C. and 850° C. are given.
3. The melting point of lead chloride has been determined as 495° C., and the boiling point as 945° C. at a pressure of 754 mm. of mercury. An average melting point of 498° C. was adopted as the best average of all determinations.
4. The vapor-pressure data are shown on a log p vs. $\frac{1}{T}$ plot and the averaged results tabulated, together with the weights of lead chloride in unit volumes of saturated vapor at 25° intervals between 500° C. and 950° C.
5. The heat of vaporization at the melting point is calculated to be 40,600 calories.
6. The vapor pressures of solid lead chloride, down to 400° C., are calculated and shown graphically.

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[a] Ehrhardt, Otto, Ueber die Bestimmung der specifischen Wärme und der Schmelzwärme bei hohen Temperaturen: Ann. Physik. Chemie, Bd. 24, 1885, p. 257.

[b] Welch, H. V., and Duschak, L. H., The vapor pressure of arsenic trioxide: Tech. Paper 81, Bureau of Mines, 1915, 20 pp.

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